

Research Article

Oxidation Resistance and Modification Reaction Mechanism of Al Coating Sprayed on Pure Ti Substrate

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Received 27 July 2018; Revised 5 September 2018; Accepted 12 September 2018; Published 15 October 2018

Academic Editor: Shuo Yin

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An Al coating was deposited on the surface of pure Ti substrate by arc spray technology. In order to enable the modification reaction between the Al coating and Ti substrate, the specimen was heated to a temperature above the melting point of Al. Oxidation testing of the uncoated Ti and coated specimen was conducted at 1073 K under an air atmosphere. The microstructure, chemical composition, and phase determination of the coatings and interfaces, before and after modification treatment, were done using SEM, EDS, and XRD methods. The relationships between the modification results and time and temperature were discussed. The results showed that, after heating at 973 K for 5 hours, there was still sufficient Al on the surface of the specimen. Only intermetallic $TiAl_3$ was formed in the diffusion region. After heating at 1073 K for 5 hours, all the Al elements diffused into the Ti substrate. Intermetallics $TiAl_2$ and Ti_3Al were also formed in the diffusion front of Al, in addition to $TiAl_3$. After heating at 1173 K for 5 hours, a new intermetallic $TiAl$ phase was formed at the interface of $TiAl_2$ and Ti_3Al . As the modification reaction time was prolonged at 1173 K, the formation of intermetallics $TiAl_2$, $TiAl$, and Ti_3Al were all increased. Among them, the formation amount of $TiAl_2 > Ti_3Al > TiAl$. The specimen after modification treatment had better high temperature oxidation resistance than the pure Ti substrate without coating.

1. Introduction

Titanium and titanium alloys are widely used in the fields of aerospace, chemical production, and the like, due to their high specific strength, low density, and the superior corrosion resistance [1–5]. However, when the temperature is higher than 873 K, the life of the materials is seriously affected due to several factors [4]. These include a decrease in the strength and decay of other mechanical properties, such as plasticity. After adding a coating that has excellent oxidation resistance to the surface of Ti alloys, the oxidation resistance was significantly improved while it maintained outstanding performance [6].

Al always has excellent oxidation resistance since a dense, stable Al_2O_3 film can be formed on the surface of

metal materials [7]. Ti-Al intermetallics are often used as the materials for heat-resistant coatings on titanium and titanium alloys due to the capability of forming Al-rich oxide scales of $TiAl$ [8]. At present, different methods have been used by many researchers to fabricate Ti-Al intermetallic coatings on alloys directly or by modification treatment, such as mechanical alloying [9], laser cladding [10], tungsten inert gas welding surfacing [11], laser surface alloying [12], high-vacuum arc ion plating [8], and high-energy ball milling that combines heat treatment [13], and a solid phase diffusion reaction [14]. However, the fabrication of an Al coating on the surface of pure Ti substrate by arc spray technology, and then heating the specimen to a temperature above the melting point of Al in order to form a $TiAl$ intermetallic, has seldom been reported [15].

In this paper, an Al coating was fabricated on the surface of a pure Ti substrate by arc spray technology. In order to enable the reaction between the Al coating and Ti substrate, the specimen was heated at different temperatures (above the melting point of Al) and times, and the impact of the modification temperature and time was determined. Oxidation testing of the specimen after modification treatment and a pure Ti control was conducted at 1073 K. The effect of the coating on the oxidation resistance, and its protection mechanism, were investigated. The results may provide some theoretical basis for the production of Ti-Al intermetallic coating by the method of modification reaction between coating and substrate at higher temperature.

2. Experimental Procedure

The substrate material used in this study was industrial pure Ti. All of the substrate specimens were formed into a 30 mm long, 30 mm wide, and 10 mm thick shape. Prior to spraying the coatings, ethanol and acetone were used to remove oil on the surfaces of substrates, and then the substrate surfaces were grit blasted with granular corundum to obtain a roughened surface. All of the surfaces of the specimens were sprayed with 0.5 mm thick Al coatings by arc spray technology, and then the specimens in different ceramic boats were treated by heating as follows under an air atmosphere in the resistance furnace: 973 K/5 h, 1073 K/5 h, 1173 K/5 h, 1173 K/10 h, and 1173 K/20 h (better results might be obtained if the heat treatment of the specimens were performed under vacuum or protection gas atmosphere). But in consideration of the practical application, the large titanium plate would be difficult to be performed heat treatment under vacuum or protection gas atmosphere). The spraying material was industrial Al welding wire (2 mm in diameter) which had a purity of greater than 99.8%. The arc spray equipment was type XDP-5, which was homemade by Shenyang University of Technology. The parameters for the arc spray process are listed in Table 1.

The microstructures of the coatings before and after modification treatment were characterized by a scanning electron microscope (SEM; S-3400 and S-4800, Hitachi, Japan). The distribution of chemical elements at the coating-interface-substrate regions was characterized by EDS. The intermetallic compounds formed in the diffusion reaction process were characterized by X-ray diffraction (XRD; Shimadzu, 7000, Kyoto, Japan) with Cu-K α radiation ($\lambda = 0.1541$ nm) at 40 KV 30 mA. Since the thickness of most Ti-Al intermetallic layers which were formed during the modification reaction was too thin, it was difficult to polish to reveal the surfaces of different layers. So in this experiment, not all the layers were identified with XRD, the chemical composition of most diffusion reaction regions was characterized by energy-dispersive spectrometer (EDS) ($V_{a/c} = 20.0$ KV), which was equipped with SEM.

The Al_2O_3 film on the specimen was removed by the waterproof abrasive paper (500#) after modification

TABLE 1: Arc-sprayed parameters.

Materials	Voltage (V)	Current (A)	Atomization compressed air supply pressure (MPa)	Distance (mm)
Al	31	180	0.6	150

treatment. It was then put in a ceramic boat, which was dried to constant weight. Oxidation resistance of the specimen after treatment was performed at 1073 K for 100 hours under an air atmosphere in the furnace. Every 10 hours, the specimen was removed from the furnace, and then it was cooled to room temperature in the atmosphere; the heating rate of the furnace was 293 K/min, and the air cooling rate was about 298 K/min. The comparative experiments for the pure Ti control were performed under similar conditions. The mass gain of the specimen was weighed during every period on an electronic balance, which was accurate to 0.1 mg. The oxidation kinetics curves of the specimen and the pure Ti were then obtained.

3. Results and Discussion

Figure 1 shows the microstructure of the interface that was not heated. As shown in Figure 1, the dark gray area is the Al coating, which was about 500 μm thick, and the region under the Al coating is the Ti substrate. There were some black pores in the coating. This occurred because after the Al particles in a molten state were sprayed on the surface of the substrate, the surface temperature of the particles decreased rapidly and lead to the large temperature difference between the surface and inside of the particles. As a result, Al particles were present as spheres and pores appeared. The porosity of the thermal spraying coating was about 3%–5%. In addition, the bonding between the Al coating and Ti substrate was mechanical. It was not very compact, and there were also some pores at the interface between the Al coating and the substrate.

Figure 2 shows the cross-sectional BSD image of the Al coating after testing for 5 hours at 973 K (Figure 2(a)) and the distributions of Al (Figure 2(b)) and Ti (Figure 2(c)). As shown in Figure 2, after heating at 973 K for 5 hours, there was still surplus Al on the surface of the specimen, and some of the Al diffused into the Ti substrate.

As shown in Figure 3, after heating at 973 K for 5 hours, a 0.5 mm thick light gray diffusion region was formed between the Al coating and the Ti substrate. The pores between the Al coating and the Ti substrate, which are shown in Figure 1, disappeared as the modification reaction proceeded. Figure 3(b) is the expanded view of region I in Figure 3(a), and it can be seen from Figures 3(a) and 3(b) that a new phase with a continuous distribution was formed in the diffusion region. The chemical composition (at.%) at points A, B, C, and D, as marked in Figure 3(b), is listed in Table 2.

It can be determined that the phases of points A, B, C, D, and E were intermetallic TiAl_3 upon comparing their chemical compositions with the theoretical ratio ranges of intermetallic TiAl_3 . The white-gray region which was

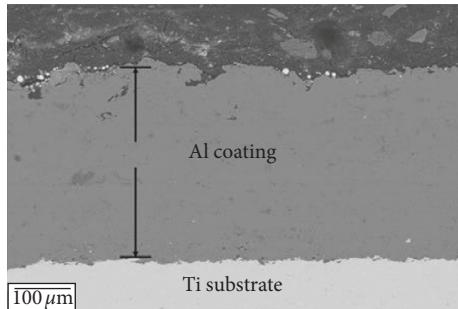


FIGURE 1: Cross-sectional BSD image of Al coating without heating.

represented by F point was Ti substrate. Since Al in the diffusion zone closed to the substrate-diffusion zone interface is not homogenous, that part was represented by A, C, and D points which looks like darker.

After heating at 973 K for 5 hours, Al coating (400–500 μm) was still on the surface of the specimen. A 500 μm thick surface layer was then removed in order to expose the diffusion region completely. The XRD results are shown in Figure 4. This data indicate that the diffusion region between the Al coating and the Ti substrate consisted of the TiAl_3 phase. This is the intermetallic structure that formed during the modification reaction between Al and Ti. Since XRD can detect phases that are typically above 2–5%, some Al might be also present on the surface of the specimen.

Figure 5 shows the cross-sectional BSD image of the Al coating after testing for 5 hours at 1073 K (Figure 5(a)) and the distributions of Al (Figure 5(b)) and Ti (Figure 5(c)). As shown in Figure 5, after heating at 1073 K for 5 hours, the Al coating disappeared and all of the Al elements diffused into the Ti substrate.

As shown in Figure 6, after the modification reaction at 1073 K for 5 hours, all of the Al diffused into the Ti substrate and a 0.9 mm thick diffusion region was formed. Compared with the thickness of the diffusion region which was formed at 973 K, the thickness increased at 1073 K. As can be seen in Figure 6, a region with different shades appeared in the diffusion region near the Ti substrate. This showed that different kinds of intermetallic compounds were formed in the diffusion front of Al. Figures 6(c) and 6(d) show the EDS results of points A, B, C, and D marked in Figure 6(b). The chemical compositions (at.%) are listed in Table 3.

After combining the data in Figures 6(c) and 6(d), along with the data in Table 3, and then comparing the chemical composition of points A, B, and C with the theoretical ratio ranges of intermetallic TiAl_3 , TiAl_2 , and Ti_3Al , it can be determined that the phases of A, B, and C were intermetallics TiAl_3 , TiAl_2 , and Ti_3Al , respectively. The 0.9 mm thick diffusion region mainly consisted of TiAl_3 phase, which was formed during the modification reaction between Al and Ti. The TiAl_2 and Ti_3Al phases were formed in the diffusion front of Al, which consumed Al and Ti gradually. The region D was Ti substrate accorded to the chemical composition.

As shown in Figures 7(a) and 7(b), after the modification reaction at 1173 K for 5 hours, the thickness of the diffusion region was about 1.1 mm, which increased compared to 1073 K. Also, the region of different shades that appeared in the diffusion front of Al, and the thickness of regions B and D, all increased compared to 1073 K. Figures 7(c) and 7(d) show the EDS results for points A, B, C, D, and E marked in Figure 7(b). The chemical compositions (at.%) are listed in Table 4.

After combining data in Figures 7(c) and 7(d), along with the data in Table 4, it can be determined that the phases of A, B, C, D, and E were intermetallics TiAl_3 , TiAl_2 , TiAl , Ti_3Al , and Ti substrate, respectively. The diffusion region still mainly consisted of TiAl_3 phase after the modification reaction at 1173 K for 5 hours. The TiAl_2 , TiAl , and Ti_3Al phases were formed in sequence in the diffusion front of Al, which advanced to the Ti substrate. Compared with the modification reaction products which were formed at 1073 K, a new TiAl phase was formed at the interface between TiAl_2 layer and Ti_3Al layer.

From the results above, it is clear that the thickness of the diffusion layer increased with modification reaction temperature, namely, the higher the temperature, the easier the diffusion from Al to the Ti substrate for the same reaction time. Furthermore, Ti changed from α to β during heating up above 1155 K, and Al is a relatively fast diffuser in β -Ti [16].

At test temperature in this paper, Ti in the substrate also diffused into Al coating. In theory, the higher the temperature, the easier the diffusion from Ti to the Al coating as well. Since Ti is a high-melting-point metal, EDS (Figure 2) showed that it was not found that there were a large number of Ti in residual Al coating.

There was still sufficient Al on the surface of the specimen after heating at 973 K for 5 hours. The obvious layer phenomenon (the interface between Ti substrate and the TiAl_3 phase) did not appear in the diffusion front of Al. The diffusion region only consisted of intermetallic TiAl_3 , according to the XRD result. In other words, only $3\text{Al} + \text{Ti} \rightarrow \text{TiAl}_3$ reaction occurred in the diffusion region when there were still sufficient Al elements on the surface of the specimen.

The Al coating disappeared and all the Al elements diffused into the Ti substrate when the modification reaction temperature was raised to 1073 K. The diffusion region still mainly consisted of TiAl_3 phase, but the layered distribution region appeared in the diffusion front of Al. The TiAl_2 and Ti_3Al phases formed in sequence in the diffusion front of Al, with decreasing Al content and increasing Ti content. In other words, the other Ti-Al intermetallic compounds were formed as the reaction proceeded between the TiAl_3 phase and Ti substrate, when there was not any Al on the surface of the specimen.

When the reaction temperature was raised to 1173 K, a new TiAl phase was formed besides the intermetallics TiAl_2 and Ti_3Al , which were layered in the diffusion front of Al. According to the result of Sujata et al. [17], $\Delta G_f \text{TiAl}_2 < \Delta G_f \text{TiAl}_3 < \Delta G_f \text{Ti}_3\text{Al} < \Delta G_f \text{TiAl} < 0$ when the temperature range is about 900–1400 K. Intermetallic TiAl was more difficult to be formed than TiAl_2 and Ti_3Al from the

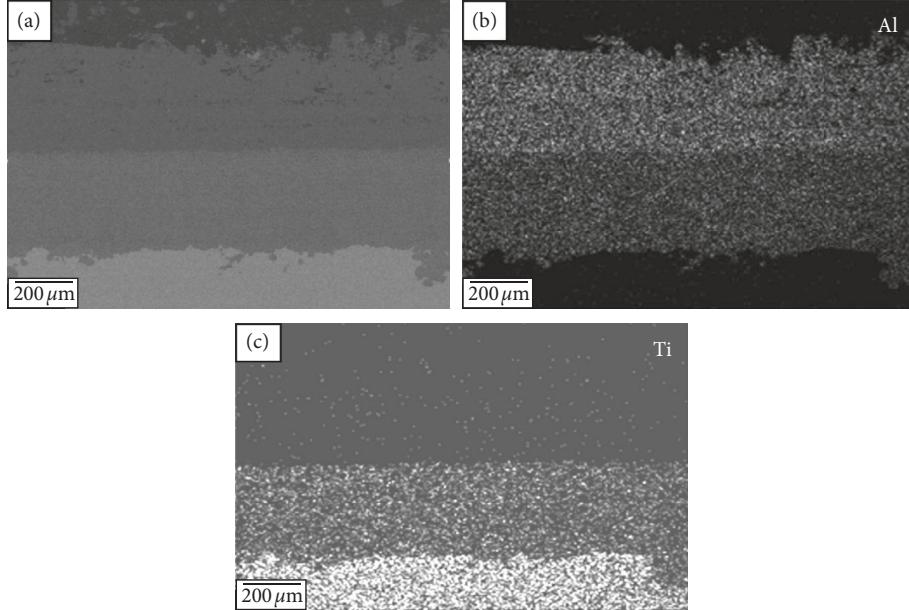


FIGURE 2: (a) Cross-sectional BSD image of Al coating after testing for 5 hours at 973 K and the distributions of (b) Al and (c) Ti.

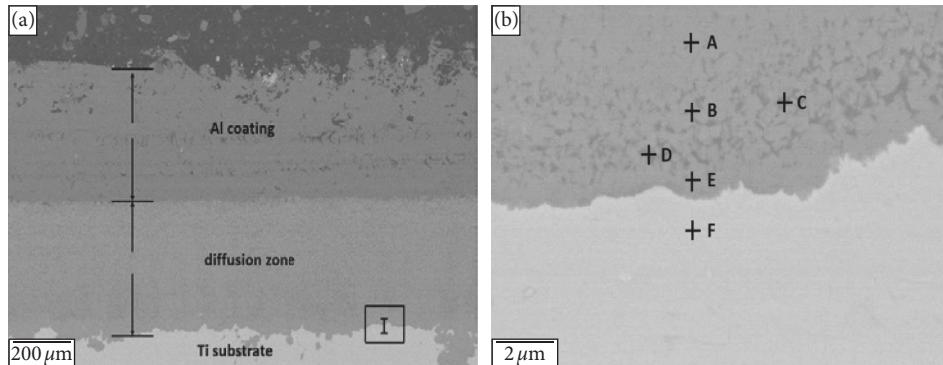


FIGURE 3: (a) Cross-sectional BSD image of Al coating after testing for 5 hours at 973 K, and (b) the expanded view of region I in Figure 3(a).

TABLE 2: The chemical composition (at.%) of different points, as marked in Figure 3(b).

Points	Al	Ti
A	77.61	22.39
B	75.28	24.72
C	78.62	21.38
D	77.79	22.21
E	74.43	25.57
F	0	100

thermodynamics, which explains why the TiAl phase formed during the modification reaction at 1173 K, but not at 1073 K.

In addition, the thickness of TiAl₂ and Ti₃Al both increased at 1173 K compared with at 1073 K. This showed that the increase in the temperature could contribute to further reaction between TiAl₃ phase and Ti substrate.

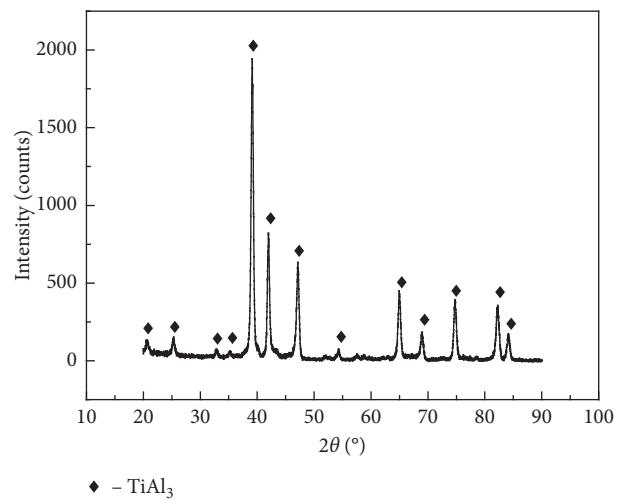


FIGURE 4: XRD spectrum after removal of 500 μm thick surface layer.

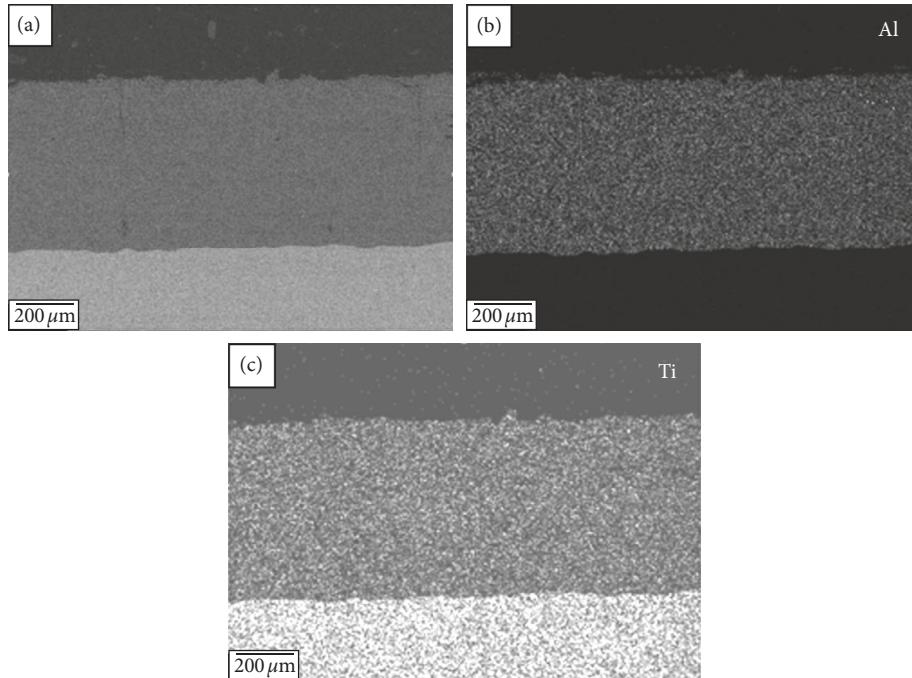


FIGURE 5: (a) Cross-sectional BSD image of Al coating after testing for 5 hours at 1073 K and the distribution of (b) Al and (c) Ti.

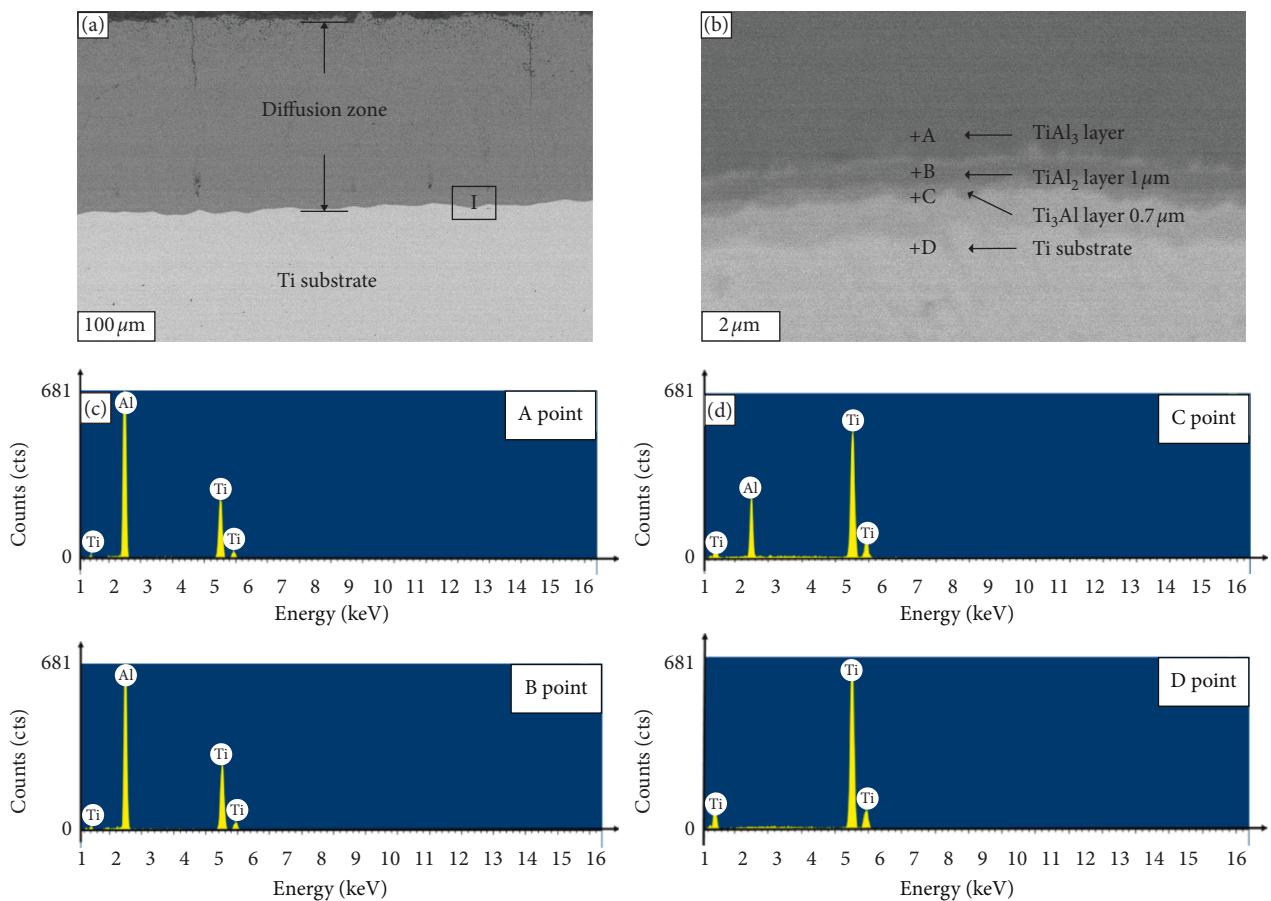


FIGURE 6: (a) Cross-sectional BSD image of Al coating after testing for 5 hours at 1073 K, (b) the expanded view of region I in Figure 6(a), (c) EDS results of points A and B in Figure 6(b), and (d) EDS results of points C and D marked in Figure 6(b).

TABLE 3: The chemical composition (at.%) in different points, as marked in Figure 6(b).

Points	Al	Ti
A	73.77	26.23
B	67.49	32.51
C	29.27	70.73
D	0	100

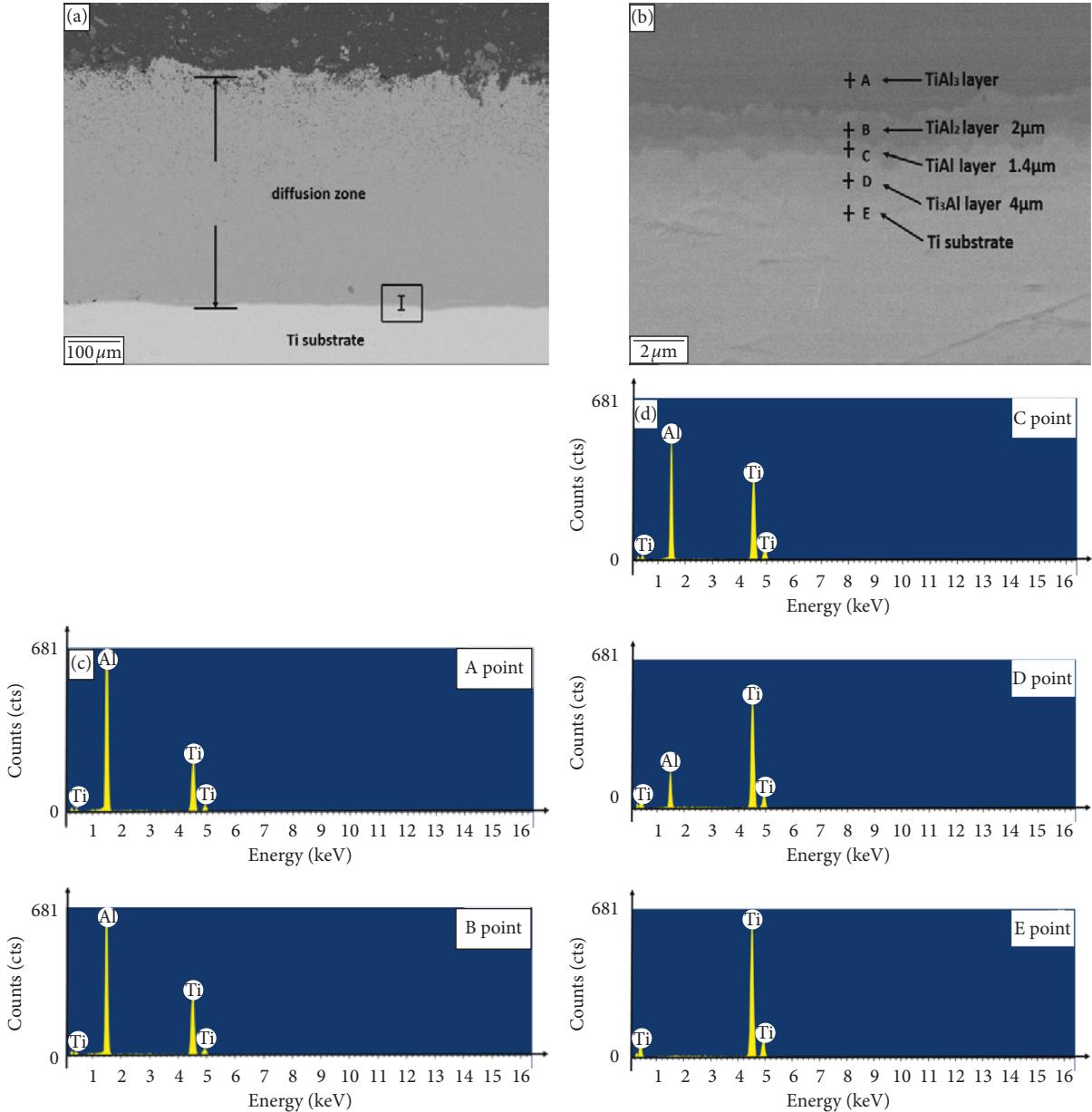


FIGURE 7: (a) Cross-sectional BSD image of Al coating after testing for 5 hours at 1173 K, (b) the expanded view of region I in Figure 7(a), (c) EDS results of points A and B marked in Figure 7(b), and (d) EDS results of points C, D, and E marked in Figure 7(b).

As shown in Figure 8(a), the microstructure of the diffusion region after modification treatment at 1173 K for 10 hours was basically identical to it after 5 hours. Figure 8(b) shows that the thickness of B, C, and D layers in the diffusion region near the Ti substrate was thicker than that under the reaction condition of 1173 K/5 hr. The largest increase in the

thickness was layer B, and the smallest one was layer C. The chemical compositions (at.%) of points A, B, C, D, and E as marked in Figure 8(b) are listed in Table 5.

As seen from the data in Table 5, the products which were formed after modification treatment at 1173 K for 10 hours, and their distributions, were not significantly

TABLE 4: The chemical composition (at.%) in different points, as marked in Figure 7(b).

Points	Al	Ti
A	74.08	25.92
B	65.12	34.88
C	52.22	47.78
D	22.08	77.92
E	0	100

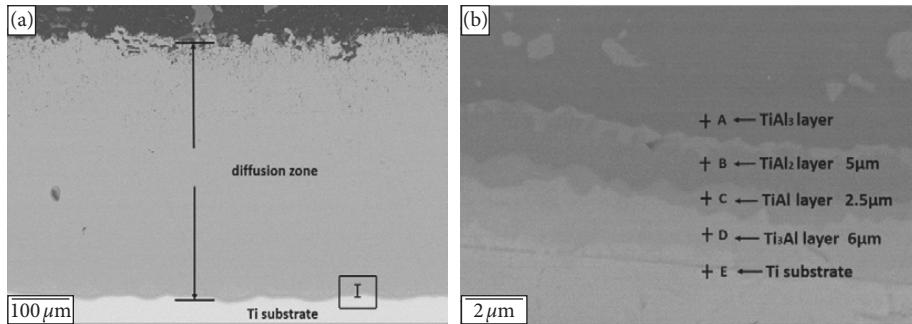


FIGURE 8: (a) Cross-sectional BSD image of Al coating after testing for 10 hours at 1173 K and (b) the expanded view of region I in Figure 8(a).

TABLE 5: The chemical composition (at.%) in different points, as marked in Figure 8(b).

Points	Al	Ti
A	74.08	25.92
B	65.37	34.63
C	48.06	51.94
D	26.42	73.58
E	0	100

changed compared with that under the reaction condition of 1173 K for 5 h. The diffusion region still mainly consisted of TiAl₃ phase and intermetallics TiAl₂, TiAl, and Ti₃Al formed in sequence in the diffusion front of Al.

As shown in Figure 9(a), the layered structure was more obvious in the diffusion front of Al after modification treatment at 1173 K for 20 hours. In addition, it can be seen in Figure 9(a) that the thickness of B and D layers increased compared with that under the reaction condition of 1173 K for 10 hours. The C layer had less change in the thickness. The chemical compositions (at.%) of points A, B, C, D, and E as marked in Figure 9(b) are listed in Table 6.

As can be seen from the data in Table 6, the products which were formed after the modification treatment of the specimen at 1173 K for 20 hours, and their distributions, were all the same with that under the reaction condition of 1173 K for 5 hours and 1173 K for 10 hours.

As shown in Figure 9(c), the layered structure in the diffusion front of Al was comprised of different kinds of Ti-Al intermetallic. It could be revealed that the element ratio of titanium to aluminum in TiAl₃ and TiAl₂ was a fixed value, but for TiAl and Ti₃Al, it was within a certain range.

The products formed after the modification treatment of the specimens at 1173 K for 10 and 20 hours, and their distributions, were the same as that under the reaction condition of 1173 K for 5 hours. The diffusion region mainly consisted of intermetallic TiAl₃, and intermetallics TiAl₂, TiAl, and Ti₃Al were formed in sequence in the diffusion front of Al, with decreasing Al content and increasing Ti content.

As can be seen from the data in Table 7, the thickness of TiAl₂, TiAl, and Ti₃Al layers were all increased as the reaction time was prolonged. This was because a concentration gradient of Ti was still present between TiAl₃ phase and Ti substrate. The Ti element in Ti substrate continuously diffused into TiAl₃ layer and reacted with it under the concentration gradient driving forces, and then intermetallics TiAl₂, TiAl, and Ti₃Al were formed. It is also apparent from Table 7 that the increased thickness of TiAl₂ layer > the increased thickness of Ti₃Al layer > the increased thickness of TiAl layer. This was because, as mentioned above, ΔG_f TiAl₂ < ΔG_f TiAl₃ < ΔG_f Ti₃Al < ΔG_f TiAl < 0 when the temperature range is about 900–1400 K. Therefore, the formation amount of TiAl₂ phase > the formation amount of Ti₃Al phase > the formation amount of TiAl phase. The growth of TiAl₂ phase and TiAl phase was in a competitive relationship, and the growth speed of TiAl₂ was 1.4 times the growth speed of TiAl [18]. This also made that the increase in the thickness of TiAl₂ layer larger than that of TiAl layer as the reaction proceeded.

In order to test the high temperature oxidation resistance of Ti-Al intermetallics which were formed during the modification reaction, 100 μm thick Al₂O₃ surface layer that occurred after the modification reaction at 1173 K for 20 hours was removed by waterproof abrasive paper to expose the TiAl₃ layer completely. Then, oxidation resistance of the Al/Ti specimen after treatment and the pure Ti without

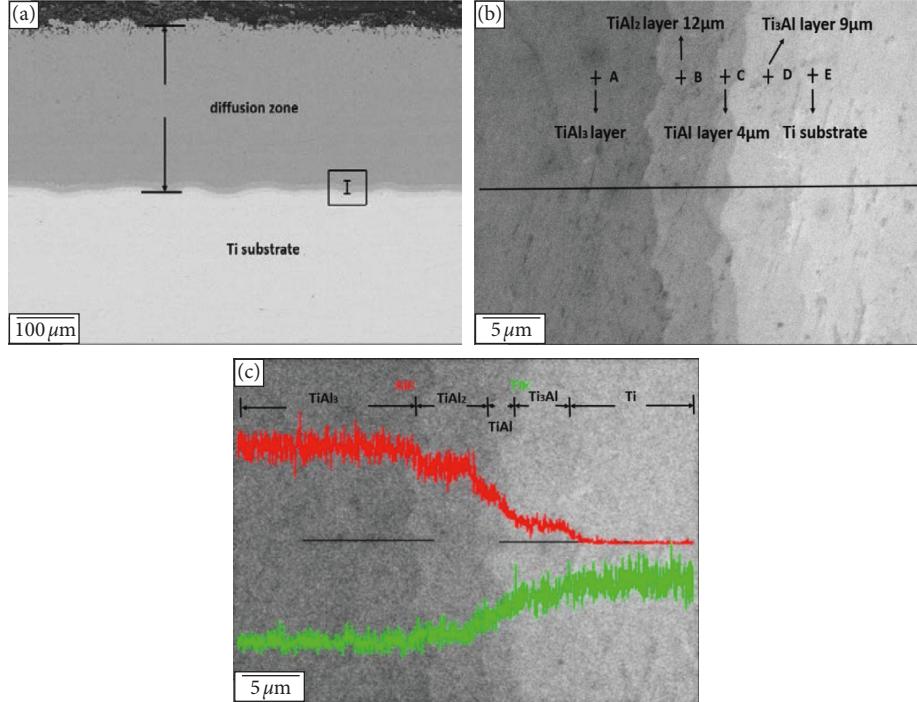


FIGURE 9: (a) Cross-sectional BSD image of Al coating after testing for 20 hours at 1173 K, (b) the expanded view of region I in Figure 9(a), and (c) concentration profiles of Al, Ti elements in Figure 9(b).

TABLE 6: The chemical composition (at.%) in different points, as marked in Figure 9(b).

Points	Al	Ti
A	74.74	25.26
B	65.65	34.35
C	50.44	49.56
D	22.04	77.96
E	0	100

TABLE 7: Thickness (μm) of different Ti-Al Intermetallics layers with 1173 K/5 h, 1173 K/10 h, and 1173 K/20 h.

Ti-Al intermetallics	1173 K/5 h	1173 K/10 h	1173 K/20 h
TiAl ₂	2	5	12
TiAl	1.4	2.5	4
Ti ₃ Al	4	6	9

coating were performed at 1073 K for 100 hours under air atmosphere.

It could be seen from the oxidizing dynamic curves in Figure 10 that the curve for the pure Ti without coating is nearly straight. This was in a high-temperature environment, so it was easy for Ti to be oxidized by molecular oxygen to produce TiO₂, which was easy to break. The TiO₂, which had a rutile structure, peeled off from the surface of the pure Ti specimen and the exposed Ti substrate to be oxidized again.

The oxygen weight gain of the Al/Ti specimen (after modification treatment and removal of the Al₂O₃ film) in the later stage of the oxidation exhibited a reduction as compared to the beginning of the oxidation. This was because intermetallics TiAl₃, TiAl₂, and others, which were rich in Al,

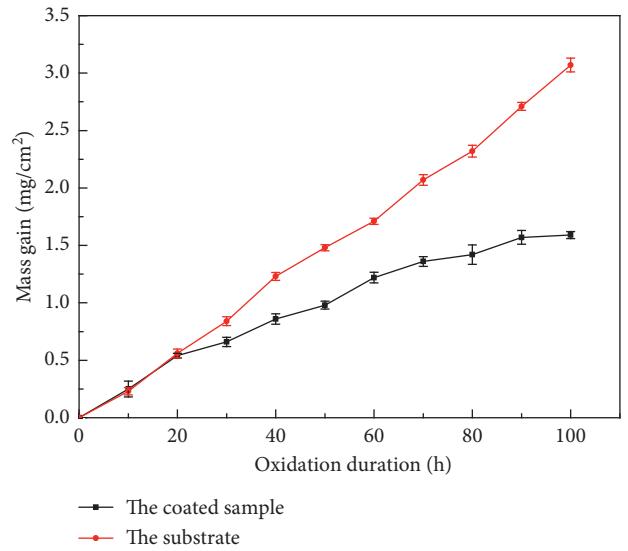


FIGURE 10: Oxidation kinetics curves of Ti alloy with and without coating at 1073 K for 100 h (10 cycles).

formed in the process of modification reaction between the Al coating and Ti substrate. The Al₂O₃ was formed by the reaction between Al-rich TiAl₃ phase and the oxygen in air at the beginning of the oxidation. The surface of the specimen was covered with Al₂O₃ film, which increased constantly. Furthermore, the oxidation of the specimen was sufficiently suppressed. But a little amount of the Al₂O₃ film peeled off from the surface of the specimen with the increase in the high temperature oxidation time, and the exposed TiAl₃

phase would be oxidized again. The weight of the specimen also increased during the later stage of the oxidation but with smaller increasing amount.

The total weight gain of noncoated Ti and coated Al/Ti specimen was 3.1 mg and 1.6 mg, respectively, after oxidation for 100 hours as it is shown in Figure 10. The total weight gain of noncoated Ti was almost twice than the coated Al/Ti specimens.

The Al/Ti specimens after modification treatment had a better high temperature oxidation resistance than the pure Ti without protection coating.

4. Conclusions

- (1) After heating at 973 K for 5 hours, there was still sufficient Al on the surface of the specimen, and only intermetallic $TiAl_3$ formed in the diffusion region.
- (2) After heating at 1073 K for 5 hours, all of the Al diffused into the Ti substrate, and intermetallics $TiAl_2$ and Ti_3Al also formed in the diffusion front of Al, in addition to $TiAl_3$.
- (3) After heating at 1173 K for 5 hours, a new intermetallic $TiAl$ phase was formed at the interface of $TiAl_2$ and Ti_3Al , in addition to the intermetallics $TiAl_2$ and Ti_3Al in the zone of the diffusion reaction.
- (4) As the modification reaction time was prolonged at 1173 K, the formation of intermetallics $TiAl_2$, $TiAl$, and Ti_3Al all increased, and among them, the formation amount of $TiAl_2$ phase > the formation amount of Ti_3Al phase > the formation amount of $TiAl$ phase.
- (5) The Al/Ti specimens after modification treatment had better high temperature oxidation resistance than the pure Ti without protection coating.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

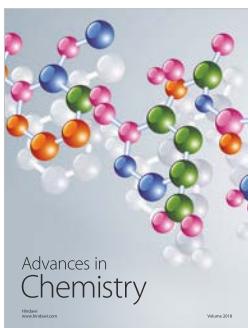
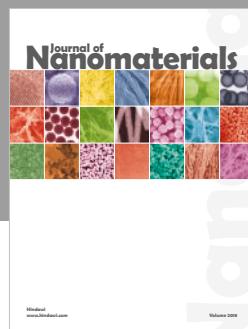
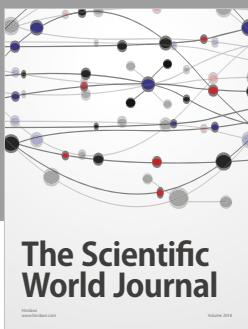
The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

This work was financially supported by the Natural Science Foundation of Liaoning Province (No. 201602553) Chinese National Natural Science Foundation (No. 51301112).

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